### Notice:

The attached document was obtained from the National Air and Radiation Environmental Laboratory, United States Environmental Protection Agency, and may be used for reference or to perform work. The controlled copy of this document resides with the NAREL Document Control Officer. This copy is not controlled. Users of uncontrolled copies, from whatever source, are personally responsible for ensuring that the most current version is used as reference or to perform work. Do not retain the printed copy to perform future work without first verifying that it is the most current version.

# NAREL STANDARD OPERATING PROCEDURE MASS MEASUREMENTS

## **FOR THE**

# ${ m PM}_{ m 2.5}$ CHEMICAL SPECIATION QA PROGRAM

#### PM2.5/SOP-1

Monitoring and Analytical Services Branch
National Air and Radiation Environmental Laboratory
Office of Radiation and Indoor Air

Revision:	1	Copy Number:		
Date:	2004-08-17	Controlled Copy: Yes	No _	Χ
Assigned to:				
DCO:				
				•

## PM2.5/SOP-1

# **REVISION HISTORY**

Rev.	Responsible Official	Date
0	Jewell Smiley	2000/09/01
1	Jewell Smiley	2004/08/17

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 1 of 13

#### 1.0 PURPOSE

1.1 This document describes the procedures used at the National Air and Radiation Environmental Laboratory (NAREL) to gravimetrically determine the mass of air filters before and after exposure to airborne particulate matter. The gravimetric analyses performed at NAREL are part of the Laboratory Quality Assurance Program for the  $PM_{2.5}$  Chemical Speciation Network.

#### 2.0 SCOPE AND APPLICATION

- 2.1 This document provides detailed instructions for the gravimetric analysis of captured particles deposited upon a 46.2-mm filter. Procedures for sample inspection, measurement, calculation, documentation, archival, safety, and quality control are included.
- 2.2 This method is restricted for use by or under the supervision of analysts experienced in the use of a microbalance. Each analyst must demonstrate the ability to generate acceptable results with this method.

#### 3.0 **DEFINITIONS**

- 3.1 **CCV** Continuing Calibration Verification. A check standard which is analyzed periodically to test the accuracy of the measurement system.
- 3.2 **COC** Chain Of Custody. An unbroken trail of accountability that ensures the physical security of samples, data, and records.
- 3.3 **EDXRF** Energy Dispersive X-Ray Fluorescence. A non-destructive analytical technique which excites the sample atoms to create temporary vacancies of inner shell electrons. The excited atoms release x-ray photons which intercept a rapid-response detector. Detector response is usually recorded using a multi-channel analyzer such that each channel represents a different x-ray energy emitted from the sample. By counting detector responses over time, a sample spectrum (counts versus photon energy) may be acquired.
- 3.4 **MASB** Monitoring and Analytical Services Branch. The branch at NAREL is responsible for the analysis of environmental samples for radioactive and/or mixed waste contamination.
- 3.5 **NAREL** National Air and Radiation Environmental Laboratory.
- 3.6 **NIST** National Institute of Standards and Technology. Metallic weights may be purchased from a vendors such as Fisher Scientific with certificates of analysis that trace the gravimetric mass measurement back to NIST measurements.

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 2 of 13

3.7 **PM**<sub>2.5</sub> - Particulate Matter with an aerodynamic diameter less than or equal to 2.5 micrometers.

- 3.8 **PTFE (Teflon®)** Polytetrafluoroethylene manufactured and marketed by Dupont using its "Teflon" registered trademark.
- 3.9 **QA Manager** Quality Assurance Manager. The person with primary responsibility for overseeing the NAREL QA/QC Program.
- 3.10 **RTI** Research Triangle Institute, the independent contractor laboratory for the  $PM_{2.5}$  Chemical Speciation Program.
- 3.11 **SHEM** Safety Health and Environmental Management
- 3.12 **SOP** Standard Operating Procedure. The officially approved document that describes in detail the steps of a procedure for performing a routine or repetitive task.
- 3.13 **XRF** X-Ray Fluorescence is a technique used to determine the elemental composition of particulate matter collected onto an air filter.

#### 4.0 SUMMARY OF METHOD

4.1 New filters are received from the vendor and visually inspected for defects before they are equilibrated within a clean environmental chamber held at constant temperature and humidity. Each filter is identified by a unique serial number permanently attached to its mounting ring. After equilibration has been achieved to reach a constant mass, each clean filter is weighed and shipped to the field for sample collection. After the sampling event is complete, the filter is returned to the laboratory and placed into the environmental chamber for re-equilibration at its initial weighing temperature and humidity. The difference in mass between the initial and final weighing is a measure of the particulate matter collected onto the filter during the field sampling event.

#### 5.0 INTERFERENCES

- 5.1 Effort must be made to prevent the accidental contamination of a filter. Each filter is scheduled for gravimetric analysis and for elemental analysis using Energy Dispersive X-Ray Fluorescence (EDXRF). Contaminants (other than moisture) accidently added to the filter may not only cause error for the mass determination, but may also cause error for the elemental analysis. Near clean room conditions and practices are maintained in the environmental weighing chamber to minimize accidental contamination of filters.
- 5.2 Effort must be made to prevent loss of material (other than moisture) from a loaded filter. After loading, filters must not be exposed to temperatures above 25 °C, excessive vibration, or abrasive scratches.
- 5.3 Weight loss due to mechanical removal of particles from the filter will be minimized by carefully removing the filter from its module. Electrostatic charge can accumulate on the

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 3 of 13

filter which may lead to loss of load particles due to electrostatic repulsions. Furthermore, any electrostatic charge still present on the filter at the balance during the actual weighing event, may cause an immediate weighting error.

5.4 Weight loss of a loaded filter may be caused by chemical decomposition or evaporation of compounds like ammonium nitrate, which releases ammonia and nitric acid as gases. Semivolatile organic compounds may be part of the particulate matter on filters, and if so, they may evaporate and cause sample weight loss. Such weight losses will be minimized by keeping the filters cool during transportation to the laboratory.

#### 6.0 EQUIPMENT AND SUPPLIES

- 6.1 Environmental weighing chamber capable of providing a dust free atmosphere with temperature and humidity control that satisfies the rigorous requirements of the program.
- 6.2 Mettler MT5 Microbalance.
- 6.3 Marble balance table.
- 6.4 Set of standard weights traceable to the National Institute of Standards and Technology (NIST).
- 6.5 Dickson Data Logger with NIST-traceable calibration for temperature and humidity measurements.
- 6.6 Computer complete with all the necessary software and hardware to interface with the balance, the chamber temperature and humidity monitors, and a spreadsheet for data collection.
- 6.7 Filters, Whatman Catalog Number 7592-004 or equivalent. Each filter is a 46.2-mm circular disk which consists of a polypropylene mounting ring around the perimeter of a PTFE (Teflon®) membrane approximately 40 µm thick with 2-µm pores. The mounting ring contains a unique serial number for each filter. Each filter lot must be pretested according to EPA requirements (40 CFR Part 50, Appendix L) for performance and cleanliness.
- 6.8 Two Refrigerators, one dedicated to temporary storage of filters not completely analyzed and one dedicated to long-term archival of filters.
- 6.9 Plastic forceps to handle filters.
- 6.10 Petrislides.
- 6.11 Portable rack with removable shallow trays to hold filters during equilibration.
- 6.12 Staticide.
- 6.13 Lint free antistatic wipe cloth.
- 6.14 Polonium strips. Po<sup>210</sup> is an alpha emitter with a half life of 138 days.
- 6.15 Powder free antistatic gloves.
- 6.16 Lab coat.
- 6.17 Shoe covers.

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 4 of 13

6.18 Sticky floor mats.

6.19 Laboratory Notebook.

#### 7.0 SAFETY

7.1 The polonium strips used to neutralize electrostatic charge possibly present on a filter are weak alpha emitters with a useful life of approximately one year. Contact the radiation safety officer at NAREL for disposal of these polonium strips as a radioactive waste.

- 7.2 All procedures performed at NAREL must be conducted following the requirements detailed in the NAREL Chemical Hygiene Plan and the NAREL Radiation Safety Manual.
- 7.3 All NAREL laboratory personnel are expected to use good laboratory practices. Most of the safety training is provided by the SHEM officer. All lab personnel are expected to conform to directives given by the SHEM officer.

#### 8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 8.1 Because of its QA role within the  $PM_{2.5}$  Chemical Speciation Network, NAREL will analyze samples that are split with one of the program laboratories. NAREL will work closely with each participating laboratory to coordinate its QA activities with the on-going laboratory services that are routinely provided to the program. The sequential events in the life cycle of a filter analyzed at NAREL are described as follows.
  - 8.1.1 After they are manufactured, new filter lots must undergo rigorous testing by the manufacturer as described in 40 CFR Part 50, Appendix L, before filters are released to the laboratory.
  - 8.1.2 Filters are received at NAREL, and each individual filter is further inspected for defects. Each filter that passes inspection is placed into a labeled petri slide.
  - 8.1.3 Clean pre-loaded filters are shipped to the participating laboratory with chain-of-custody (COC) documentation. The participating laboratory determines a tare mass for each filter and then ships the filters back to NAREL.
  - 8.1.4 After the filters are received back at NAREL, they are immediately placed into the environmental chamber for equilibration and weighing to independently determine a tare mass at NAREL.
  - 8.1.5 After the tare mass has been determined at both laboratories, each filter is assembled into a sampler module and loaded with  $PM_{2.5}$  captured from the ambient air.
  - 8.1.6 After the collection event has finished, the exposed filter is removed from its module and placed back into NAREL's environmental chamber for equilibration and

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 5 of 13

re-weighing to determine its catch. A capture value is calculated for each filter only after a very stable mass has been demonstrated by weighing each filter multiple times.

- 8.1.7 After NAREL has finished its analysis, the loaded filters are shipped [cool] back to the participating laboratory using express mail. The loaded filters must be weighed again at the participating laboratory using their standard procedures. After all of the analyses are complete, the sample set is usually shipped back to NAREL and placed into archive.
- 8.1.8 Results from the participating laboratory are compared to the results determined at NAREL. Sample sets may contain blank filters as well as loaded filters and may furthermore contain metallic weights that are measured at both laboratories.
- 8.2 Routine sample collection is a field activity, and the details of field activities are beyond the scope of this SOP. Field personnel typically receive each filter already mounted in a module appropriate for the air sampler at that site. Modules are attached to the air sampler. A grab sample from the ambient air is collected onto the filter usually for a period of 24 hours. After sample collection, the filter module is removed from the sampler, and the entire module is shipped back to the laboratory for analysis.
- 8.3 Samples collected in the field are preserved by cooling to 4 °C. After analysis in the laboratory, filters are returned to 4 °C for archival.
- 8.4 NAREL will provide Chain-Of-Custody (COC) documentation with all sample shipments and transfers to ensure that samples are handled by authorized personnel. A written record is maintained of each sample from the time of its collection through preparation, analysis, and archival.

#### 9.0 PROCEDURE

- 9.1 New filter lots received at NAREL will be logged into a filter inventory database used to track the activities of each individual filter. Additional testing, beyond that already completed by the manufacturer, will be performed as follows.
  - 9.1.1 Each individual filter will be examined using a light box and magnifying lens for visual signs of manufacturing flaws. A filter will be rejected for use in the program if it contains any of the following defects.
    - 9.1.1.1 Pinholes.
    - 9.1.1.2 Separation of ring
    - 9.1.1.3 chaff or flashing
    - 9.1.1.4 loose material
    - 9.1.1.5 discoloration

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 6 of 13

9.1.1.6 filter nonuniformity

9.1.1.7 any other observed defect which might degrade filter performance

- 9.2 Use the environmental chamber to equilibrate each new filter to a constant weight.
  - 9.2.1 The chamber is controlled to provide a clean working environment with a constant 21 °C (70 °F) temperature and constant 35 % relative humidity. A data logger records the temperature and humidity of the chamber every minute of every day, and these data are downloaded onto a network drive for archival at least once per week. Each download will be saved in a file using the following naming convention.

mmddyy.spl (example: g:\pm2\_5\dickson\070400.spl for data downloaded on July 4, 2000)

- 9.2.2 Information is collected for each new lot of filters to provide an estimate of the time necessary for new filters to reach constant mass in the chamber. The initial lot stability test is conducted as follows.
  - 9.2.2.1 Select two filter boxes from the same filter lot.
  - 9.2.2.2 Randomly select three filters from each box.
  - 9.2.2.3 Place the filters in petrislides and place the lid loosely over the petrislide covering approximately 75% of the filter. Allow the filters to condition overnight.
  - 9.2.2.4 Weigh the six filters and return them to their petrislides to further condition overnight. Weigh the filters again.
  - 9.2.2.5 Continue the cycle of weighing and conditioning for at least five days and plot the trend of weight loss. If the trend is still decreasing (or increasing) mass, continue the schedule of overnight conditioning and weighing until the filters have reached a stable mass. A sufficiently stable mass has been reached if the overnight change in mass is less than 5  $\mu g.$
  - 9.2.2.6 Once the filter weights stabilize, the filters are considered equilibrated.
  - 9.2.2.7 Record the length of time it took the filters to equilibrate. This will be the time that all filters from this lot must condition prior to performing a batch stability test described in the following paragraph. All data from the initial lot stability test must be stored in the laboratory database.
- 9.2.3 Each batch of new pre-loaded filters must be equilibrated in the environmental chamber and weighed before it is ready for subsequent sample collection. A batch stability test is performed to weigh and record the mass of each individual filter and verify that every filter in the batch has reached mass equilibrium before it is used for subsequent sample collection. A new pre-loaded filter is not equilibrated until the measured mass determined on separate days differs in value by less than 5 µg. The following steps will be followed during the batch stability test.
  - 9.2.3.1 Determine the number of filters that need for the next project and place that number of filters into the chamber for conditioning. All filters should be from the

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 7 of 13

same lot.

9.2.3.2 Consult the initial lot stability test results and leave the batch of filters in the chamber for the predicted number of days to equilibrate.

9.2.3.3 Start a weighing session by first examining the chamber temperature and humidity conditions for the previous 24-hour period. Download the most recent data from the Dickson data logger. For the previous 24-hour period, the mean temperature must be 20-23 °C (68.0-73.4 °F) and the mean relative humidity must be 30-40 %. Furthermore during the previous 24-hour period, there must be no temperature excursions outside 20-23 °C (68.0-73.4 °F), and there must be no excursion of relative humidity outside 30-40 %. Failure to meet these criteria for chamber conditions prevents a valid weighing session and corrective actions must be taken to bring the chamber back into control. If all chamber criteria are satisfied, the weighing session may proceed.

9.2.3.4 Open the Lotus  $123^{\odot}$  template (see Figure 1 for an example of the template). Enter the chamber conditions for the previous 24-hour period including the mean, maximum, and minimum statistics for both temperature and relative humidity. Enter the Laboratory ID of all samples to be weighed during the session. Also enter the name of the session into the template. The session name and the file name shall be the same, and the following naming convention should be used to create the session/file name.

Mmm ddyyA.123 (example: g:\pm2\_5\mass\M070400A.123 for the first weighing session performed on July 4, 2000)

The naming convention begins with an "M" for Mass, followed by six characters expressing the date of the session, and ends with an "A" for the first session of the day, a "B" for the second session, and so forth.

- 9.2.3.5 Electrical power should not be removed from the balance during its standby mode. Activate the balance and perform a calibration check using the two 2.5-gram internal weights. If the check indicates more than 1  $\mu g$  of drift, re-calibrate the balance using the internal weights. If re-calibration is ineffective, notify the supervisor to take corrective action.
- 9.2.3.6 Start the BalanceLink® software and position the cursor in the open Lotus 123® spread sheet to record the first mass measurement.
- 9.2.3.7 Every valid weighing session must begin, continue, and terminate with Continuing Calibration Verification (CCV) checks. NIST-traceable 200-mg, 100-mg, and 2-mg standards are used for this purpose. All three CCV denominations must be weighed at the beginning and at the end of the session. During the session, the 100-mg CCV must be re-weighed after every ten filters. Each CCV measurement must be compared to the historical true mass of the standard as recorded in the spread sheet. All CCV measurements must be within 3  $\mu$ g of its historical true value. Failure of any CCV requires re-analysis of all filters since the last successful calibration check.

Note: The historical true mass of a CCV check standard must be established experimentally at NAREL by weighing each standard twice on three separate days. If all six determinations are within 3  $\mu g$  of the calculated mean, then the mean value is accepted as the historical true

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17

Page: 8 of 13

mass. If any of the six determinations do not meet criteria, then the measurement system is out of control and corrective action must be performed before the historical true mass is re-evaluated.

9.2.3.8 Every weighing session must include at least one laboratory blank. Each individual filter going to the field must have a single laboratory blank associated with it. A laboratory blank may be assigned to a filter only after demonstration that its mass is sufficiently stable. The mass of a laboratory blank is not sufficiently stable until the measured mass determined on separate days differs in value by less than 5  $\mu$ g. One laboratory blank may be associated with more than one filter going to the field.

Note: Laboratory blanks are kept equilibrated inside the chamber and weighed repeatedly. They are an indicator of chamber cleanliness as well as overall weighing technique. The laboratory blank must be re-weighed each time an associated filter returns from the field. A flag must be applied to the field sample if the associated laboratory blank fails to re-weigh within 15 µg of its previous value recorded at the pre-loaded filter session.

- 9.2.3.9 For every weighing session, each filter must be weighed twice. The first weight is not confirmed by the second determination if the two values differ by more than 5  $\mu$ g. Failure to confirm an original weight requires additional measurement(s) until a reliable mass is confirmed. Failure to confirm a reliable mass of a single preloaded filter indicates a problem with that specific filter, and it should be discarded. Failure to confirm <u>multiple</u> pre-loaded filters indicates a problem with the measurement system and requires corrective action.
- 9.2.3.10 Use forceps to handle the filters and the CCV standards. Each filter must be placed upon the polonium strips for at least 60 seconds before it is transferred onto the balance pan.
- 9.2.3.11 Zero the balance if necessary. Open the door and place the first sample onto the center of the balance platform. Wait for the balance to display a stable reading, and then press the print button to send the balance reading into the spread sheet.
- 9.2.3.12 Remove the sample from the balance and shut the door. The balance reading should return to zero on its own. If after several seconds the balance has a steady reading above or below zero, press the tare key. It should not be necessary to re-zero the balance after every sample.
- 9.2.3.13 Continue the same process to weigh all samples (filters and CCVs). After all samples have been successfully weighed, save the Lotus  $123^{\text{@}}$  spreadsheet and return the balance to its standby mode.
- 9.3 The clean pre-weighed filtermust be assembled into an appropriate sampler module before it is ready for deployment to a predetermined field site.
- 9.4 After the air sampling event at the field site, filters will be returned to NAREL still mounted in the sampler module. The COC will be carefully examined for accuracy, clarity, and completeness. Field personnel will be contacted immediately to acknowledge receipt of the filter samples and to resolve any questions regarding the COC.

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17

Page: 9 of 13

9.5 Exposed (loaded) filters returned to NAREL will be removed from the sample module, inspected, placed into a petrislide, and temporarily stored inside a refrigerator or immediately placed into the environmental chamber to begin re-equilibration at a controlled temperature and humidity. After equilibration, the loaded filter mass will be determined.

- 9.5.1 The first weighing of loaded filters may be conducted after overnight conditioning in the chamber. A loaded filter is not equilibrated until the measured mass determined on separate days differs in value by less than 15 µg.
- 9.5.2 All QC procedures described above for weighing new pre-loaded filters shall apply to weighing loaded filters returned to the laboratory following the sample collection event. The following additional QC data must be recorded when loaded filters are weighed.
  - 9.5.2.1 After equilibration, a loaded filter should not weigh less than its pre-loaded value. Record a flag comment for any loaded filter that weighs  $5\,\mu g$  less than its pre-loaded value.
  - 9.5.2.2 For every weighing session, each filter must be weighed twice. The first weight of a loaded filter is not confirmed by the second determination if the two values differ by more than 5  $\mu$ g. Failure to confirm an original weight requires additional measurement(s). Some loaded filters may demonstrate poor mass stability even within a single weighing session and may not indicate a problem with the measurement system. Any loaded filter that clearly demonstrates poor mass stability after multiple attempts to confirm a stable mass, will receive a flag comment.
  - 9.5.2.3 Each filter returned from the field must be re-weighed within the same session as its associated laboratory blank. Failure of the laboratory blank to reweigh within 15  $\mu$ g of its previous value recorded at the pre-loaded filter session, shall require a flag comment for the field sample.
  - 9.5.2.4 Returning field blanks must be examined for excessive contamination. Record a flag comment for any equilibrated field blank that weighs more than 30  $\mu g$  above its pre-loaded value.
  - 9.5.2.5 New pre-loaded filters were screened for defects and were discarded if defects were observed. A pre-loaded filter will also be discarded if it is affected by a laboratory accident such as dropping it on the floor. A considerable investment is associated with each loaded filter, however, and it should not be automatically discarded for observed flaws nor for minor laboratory accidents. Instead, an appropriate flag comment shall be recorded for the affected filter, and analys is of the filter shall proceed.
- 9.6 After the loaded filters have been weighed and the appropriate QC has been completed, the lids will be placed on the petrislides and the filters placed into numerical order in a Millipore slide holder. The holders will be labeled with the appropriate range of filter numbers and then placed into a plastic bag. Filters are then stored at 4 °C or colder. The archive date and location will be recorded in the database.

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 10 of 13

#### 10.0 CALCULATIONS

10.1 Calculate the mass of particulate matter ( $\mu$ g) collected onto each filter by subtracting the filter pre-loaded mass ( $\mu$ g) from the loaded mass ( $\mu$ g).

$$PM = M_{post} - M_{pre}$$

10.2 The Volume (m³) of air sampled can be calculated from field data by multiplying the elapsed sampling time (min) times the average flow rate (L/min) times a unit conversion factor (0.001 m³/L).

Volume = 
$$(Time)(Flow)(0.001 \text{ m}^3/\text{L})$$

10.3 The concentration  $(\mu g/m^3)$  of particulate matter in the air may be calculated by dividing the mass of particulate matter  $(\mu g)$  by the Volume  $(m^3)$  of air sampled.

#### 11.0 QUALITY ASSURANCE

11.1 A 100-mg and a 200-mg Class 1 mass reference standard traceable to the National Institute of Standards and Technology (NIST) will be weighed at the beginning, after every tenth sample, and at the end of each weighing session, with weights not to differ by more than 3 µg from the historical true value. If this tolerance is exceeded, all filters not bracketed by acceptable calibration checks will be re-weighed.

Note: The historical true mass of a reference standard must be established experimentally at NAREL by weighing each standard twice on three separate days. If all six determinations are within 3 µg of the calculated mean, then the mean value is accepted as the historical true mass. If any of the six determinations do not meet criteria, then the measurement system is out of control and corrective action must be performed before the historical true mass is re-evaluated.

- 11.2 Standard weight measurements will be monitored by QC charts throughout the year to determine whether any bias has developed in the weights or the balance.
- 11.3 Replicate weighing will be made of every filter at the end of each weighing session. The second weighing will be performed to confirm the filter mass determined earlier in the session. If any confirmation differs by more than 5  $\mu$ g from the first weighing, corrective action will be taken that includes re-weighing the affected filter.
- 11.4 At least one laboratory blank will be weighed during each weigh session, and its measured mass must not to differ by more than 15 µg from its initial stabilized value determined in a previous weighing session. If this tolerance is exceeded, corrective action will be taken that includes weighing additional laboratory blanks.
- 11.5 Field blanks will be used to monitor contamination during sampling, transport,

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 11 of 13

storage, or analysis. If the post sampling mass of any field blank exceeds its tare mass by more than 30  $\mu$ g, and all laboratory blanks are within criteria, then field personnel and RTI will be notified of possible contamination in the field.

- 11.6 The microbalance will be serviced and re-certified by a qualified technician at least annually.
- 11.7 The primary mass reference standards will be re-certified annually against a NIST-traceable mass standard.
- 11.8 The accuracy of the temperature and relative humidity sensors will be re-certified annually.

#### 12.0 REFERENCES

- 12.1 Reference method for the determination of fine particulate matter as PM<sub>2.5</sub> in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L. 1997.
- 12.2 Quality Assurance Guidance Document 2.12; Monitoring PM<sub>2.5</sub> in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. Environmental Protection Agency. Office of Research and Development, Research Triangle Park, NC. 1998.
- 12.3 Quality Assurance Project Plan for the Quality Assurance Laboratory Role in the Particulate Matter ( $PM_{25}$ ) Chemical Speciation Project.

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 12 of 13

#### 13.0 APPENDICES

13.1 The following table 1 summarizes QC checks performed at NAREL during the gravimetric determination of air filters analyzed for the  $PM_{2.5}$  Chemical Speciation Network.

## Table 1

Type of QC	Description	Acceptance Criteria	
Mass reference standards	Class 1 weights measured before, during, and at the end of every weighing session.	measured difference from historical true value < 3 µg	
Lot stability test	Six filters are repeatedly weighed to determine the necessary equilibration time for filters of the same lot.	< 5 µg difference between sessions and no slowly increasing or decreasing trend observed.	
Replicate weighing of filters	Re-weigh every filter at the end of every session to confirm the initial measurement. This requirement applies to sessions for pre-loaded filters and sessions for loaded filters.	< 5 µg difference between accepted measurement and confirmational value.	
Batch stability test	Every new pre-loaded filter is equilibrated and weighed on separate days to verify constant mass.	< 5 µg difference between two sessions	
Equilibration of loaded filters	Every loaded filter is equilibrated and weighed on separate days to verify constant mass	< 15 µg difference between two sessions	
Laboratory blank filters	Every field sample is associated with a laboratory blank which never leaves the laboratory and must be included in each critical weighing session of its associated field sample(s).	< 15 µg difference between tare session and session for loaded filters.	
Field blank filters	Unexposed filters from the original lot are designated as field blanks by field personnel.	< 30 µg difference between tare session and session for loaded filters.	
Laboratory holding times	${\rm HT_{pre}}$ is time between weighing the pre-loaded filter and the sampling event. ${\rm HT_{post}}$ is the time between the receipt of the loaded filter and re-weighing the loaded filter.	HT <sub>pre</sub> < 30 calendar days HT <sub>post</sub> < 10 business days	
Field duplicates	Field duplicates may be split with RTI or may be analyzed at the same laboratory.	criteria to be determined	

Document: PM2.5/SOP-1

Revision: 1

Date: 2004-08-17 Page: 13 of 13

13.2 The following figure 1 shows an example using the lotus template to record raw mass data during a tare session.

# Figure 1

Tare Session						
session start date session start time	M082200A 08/22/00 09:01 AM ib 99357	Save file as: q	Ypm2	5\mass\mass\mass\mass\mass\mass\mass\mas	= 38.7 = 36.7 = 35.1 = 70.1 = 69.5 = 69.3	OK OK OK OK
	ma				= 69.3	ŏĸ
Sam ple ID 2 ma 100 ma 200 ma 10113424 10113425 10113450 10113451 10113452	Mass 1996 100.005 200.005 140.452 142.228 141.815 138.119 141.145 140.546	09:39:50 AM	LB LB LB Final Final	Com m ents	0 K 0 K 0 K 0 K 0 K 0 K	
Confirm ations 100 mg 100 mg 10113424 10113425 10113450 10113451 10113452	Mass 100.001 140.451 142.226 141.815 138.118 141.143 140.546	7 im e 09:41:17 AM 09:43:26 AM 09:43:36 AM 09:47:46 AM 09:49:55 AM 09:52:05 AM 09:52:05 AM		Comments	Differ e 0 K - 0 001 - 0 002 0 000 - 0 001 - 0 000	
2 ma 100 ma 200ma	1.996 99.999 200.005	09:56:24 AM 09:57:50 AM 09:59:17 AM			OK OK OK	